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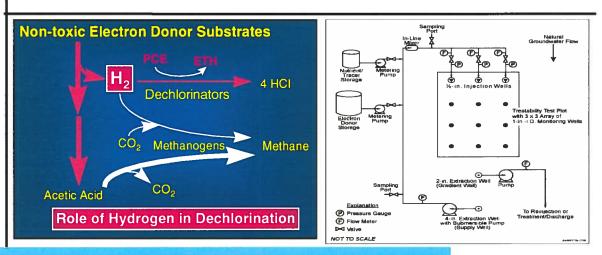
# Treatability Test for Enhanced In Situ Anaerobic Dechlorination

Problem: No testing procedure is validated for implementing enhanced reductive anaerobic dechlorination for remediation. The Air Force still has over 600 sites contaminated with chlorinated solvents such as PCE and TCE.

**Objective:** Facilitate a transition from research to full-scale implementation. Develop a cost-effective, active approach for destroying chlorinated solvent contaminants

**Technical Challenge:** Design a comprehensive study to include site specific field parameters and differences in microbial population to achieve desired level of dechlorination

Deliverable: Test protocol for implementing enhanced anaerobic dechlorination. Tool for site managers and system designers to assist in evaluating the applicability of enhanced in situ dechlorination.



Reductive Anaerobic Biological In Situ Treatment Technology (RABITT) Protocol

# **AFRL-ML-TY-TR-2001-0017**



# Reductive Anaerobic Biological In-Situ Treatment Technology (RABITT) Treatability Test

# Interim Report

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Cornell University
School of Civil and Environmental Engineering
Ithaca, New York, 14853

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# Reductive Anaerobic Biological In-Situ Treatment Technology (RABITT) Treatability Test Interim Report



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19 January, 2001

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### I. Introduction

Chloroethene compounds, such as tetrachloroethene (PCE) and trichloroethene (TCE), have been widely used for a variety of industrial purposes. Past disposal practices and accidental spills have led to widespread contamination at U.S. Department of Defense (DoD) and industrial facilities. Enhanced anaerobic dechlorination is a promising treatment approach for remediating chlorinated ethene contaminated groundwater *in situ*. The goal of this effort is to develop and validate a comprehensive approach for conducting a treatability test to determine the potential for applying reductive anaerobic biological *in situ* treatment technology (RABITT) at any specific site. A treatability protocol has been written (Morse et al., 1998) and is being applied to four DoD chlorinated solvent contamination sites in the United States. The protocol will be revised as needed upon completion of the effort based on lessons learned and field test results.

### II. Background

Because both PCE and TCE are stable compounds that resist aerobic degradation or require the presence of an electron-donating co-contaminant for anaerobic transformation, these compounds tend to persist in the environment. However, in reductive systems, highly oxidized contaminants (e.g., PCE) can be utilized as electron acceptors. RABITT attempts to stimulate this reductive pathway by supplying excess substrate (electron donor) to the native microbial consortium. The presence of the substrate expedites the exhaustion of any naturally occurring electron acceptors. As the natural electron acceptors are depleted, microorganisms capable of discharging electrons to other available electron acceptors, such as oxidized contaminants, gain a selective advantage.

The reductive dechlorination of PCE to ethene proceeds through a series of hydrogenolysis reactions, with each reaction becoming progressively more difficult to carry out. The selection of an appropriate electron donor may be the most important design parameter for developing a healthy population of microorganisms capable of dechlorinating PCE and TCE. Recent studies have indicated a prominent role for molecular hydrogen (H<sub>2</sub>) in the reductive dechlorination process (Holliger et al., 1993; DiStefano et al., 1992; Maymo-Gatell et al., 1995; Gossett et al., 1994; Zinder and Gossett, 1995). Most known dechlorinators can use H<sub>2</sub> as an electron donor, and some can only use H<sub>2</sub>. Because more complex electron donors are broken down into metabolites and residual pools of H<sub>2</sub> by other members of the microbial community, they may also be used to support dechlorination (Fennell et al., 1997; Smatlak et al., 1996; DiStefano et al., 1992).

When designing an enhanced bioremediation approach, the rate and quantity of  $H_2$  available to a degrading consortium must be considered so as to limit competition for hydrogen from other microbial groups, such as methanogens and sulfate-reducers. Competition for  $H_2$  by methanogens is a common cause of dechlorination failure in laboratory studies. As the methanogen population increases, the portion of reducing equivalents used for dechlorination quickly drops and methane production increases (Gossett et al., 1994; Fennel et al., 1997). The use of slowly degrading nonmethanogenic substrates could help prevent this type of system shutdown.

Because of the complex microbial processes involved in anaerobic dechlorination, thorough site characterization and laboratory microcosm testing are an important part of the RABITT protocol. The protocol presents a phased or tiered approach to the treatability test, allowing the user to screen out RABITT in the early stages of the process to save time and cost. The protocol guides the user through a decision process in which information is collected and evaluated to determine if the technology should be given further consideration. RABITT would be screened out if it is determined that site-specific characteristics, regulatory constraints, or other logistic problems suggest that the technology will be difficult or impossible to employ, or if competing technology clearly is superior.

## III. Approach

A summary of the protocol approach is presented here. For detailed information on sample collection techniques or analytical methods, please refer to Morse, et al. 1998.

The first phase of the treatability test includes a thorough review of existing site data to develop a conceptual model of the site. The protocol contains a rating system that can be used to assess the suitability of a site for RABITT testing. The rating system is based on an analysis of the contaminant, hydrogeologic, and geochemical profiles of the site. The decision to proceed with the RABITT screening process should be supported by data indicating that the site meets requirements for successful technology application.

The second phase of the approach involves selecting a candidate test plot location within the plume for more detailed site characterization. Characterization activities will examine contaminant, geochemical, and hydrogeologic parameters on a relatively small scale to determine the suitability of the selected location as a RABITT test plot.

Based on the information generated during the characterization of the test plot, a decision is made to proceed to phase three of the treatability study, which consists of conducting laboratory microcosm studies. Soil cores collected from the selected testing location are used to construct microsms, but prior to microcoms setup cores are visually examined to assess soil type and stratigraphy. In addition, soil core subsamples are sent to an off-site laboratory and analyzed for VOCs, TOC, and Total Iron. The microcosm studies are conducted to determine what electron donor/nutrient formulation should be field-tested to provide optimum biological degradation performance. Yeast extract, propionate, lactate, butyrate, and lactate/benzoate are evaluated for their effectiveness at stimulating anaerobic dechlorination. If the results from the microcosm testing indicate that reductive dechlorination does not occur in response to the addition of electron donors and/or nutrients, the technology is eliminated from further consideration.

The fourth and final phase of the treatability test entails field testing the electron donor/nutrient formulation determined in the laboratory microcosm tests to be most effective for supporting biologically mediated reductive dechlorination. A small test plot is set up for the addition of electron donor to the plot\_in situ. The plot is then monitored for evidence of effective dechlorination. Groundwater samples are analyzed for the following parameters: dissolved oxygen, temperature, pH, Fe<sup>+2</sup>, conductivity, chloroethenes, dissolved organic carbon, ammonia,

CH<sub>4</sub>, C<sub>2</sub>H<sub>4</sub>, C<sub>2</sub>H<sub>6</sub>, NO<sub>3</sub>, NO<sub>2</sub>, SO<sub>4</sub>, Cl, Br, alkalinity, and total iron. Table 1 presents the performance monitoring parameters and their measurement frequency during field-testing.

	e 1. List of Analytes and Fi			
Analysis	Method	Testing Location	Frequency of Analysis	
Dissolved Oxygen	DO probe	Field	Every 2 weeks	
Temperature	Temperature probe	Field	Every 2 weeks	
pН	pH probe	Field	Every 2 weeks	
Fe <sup>+2</sup>	Hach test kit	Field	Every 2 weeks	
Redox potential	Redox probe	Field	Every 2 weeks	
Chloroethenes	SW 846 Method 8260B	Laboratory	Every 2 weeks	
Dissolved Organic Carbon	EPA Method 415.1	Laboratory	Monthly	
Volatile Fatty Acids	RSKSOP-177	Laboratory	Monthly	
NH <sub>3</sub>	EPA Method 350.2	Laboratory	Monthly	
CH <sub>4</sub> , C <sub>2</sub> H <sub>4</sub> , C <sub>2</sub> H <sub>6</sub>	SW 3810 modified or Kampbell et al., 1989	Laboratory	Monthly	
NO <sub>3</sub> , NO <sub>2</sub> , SO <sub>4</sub>	EPA Method 300	Laboratory	Monthly	
Cl, Br	EPA Method 300	Laboratory	Monthly	
Conductivity	EPA Method 120.1	Laboratory	Monthly	
Alkalinity	EPA Method 310.1	Laboratory	Monthly	
pH	EPA Method 150.1	Laboratory	Monthly	

The standard RABITT field treatability test design consists of an extraction/amendment/ reinjection system within a small test plot. Contaminated groundwater is extracted near the end of the treatment plot, amended with nutrients and/or electron donor, and then reinjected near the head of the treatment plot. This design creates a hydraulic gradient to direct the flow of groundwater through the treatment plot. Monitoring points are placed within the treatment plot, in between the injection and extraction wells. Groundwater extraction and injection are optimized to achieve approximately a 30-day hydraulic residence time within the treatment plot.

The data from this phased treatability test will be evaluated to determine the potential for successfully applying RABITT in the field. The entire project effort consists of developing a draft protocol, applying the protocol at four chlorinated solvent contaminated sites and then revising the protocol based on the results observed at the four demonstration sites. To date, field treatability tests have been completed at Cape Canaveral Air Station, FL and Naval Air Station Alameda, CA. Field treatability testing was initiated at Ft Lewis, WA in August 2000, and Microcosm core samples were collected at Marine Corps Base, Camp Lejeune, NC in November 2000.

## IV. Technical Concept

- 1. Site #1: Cape Canaveral Air Station, FL
- a. Site Description:

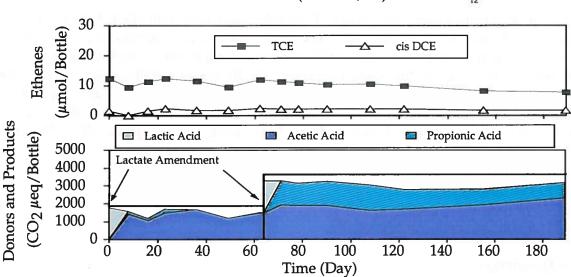
Facility 1381, the Ordnance Support Facility at Cape Canaveral Air Station, contains a shallow, 110-acre volatile organic contaminant (VOC) plume consisting primarily of TCE, DCE and VC. Improper disposal of solvents used for cleaning and degreasing operations contributed to this groundwater contamination plume. Field data suggest that TCE is naturally being dechlorinated to DCE and subsequently to VC. Each of these contaminants has been detected in a surface water body adjacent to the site.

The geology at the site is characterized by poorly sorted coarse to fine sands and shell material from ground surface to approximately 35 ft below ground surface (bgs). From approximately 35 ft to 50 ft bgs, sands show a decrease in grain size and the silt and clay content increases. From 48.5 ft to 51 ft bgs, a continuous clay unit appears to underlie the entire area at Facility 1381. Groundwater at the site is very shallow, generally ranging between 4 and 7 ft bgs. The hydraulic conductivity for the shallow groundwater has been determined to be approximately 88.7 ft/day. The pH of the groundwater ranged from 6.87 to 8.14 and conductivity readings ranged from 464 to 5,550 umhos/cm. The groundwater flow velocity has been calculated to be 0.21 ft/day. The suspected source area contains high levels of TCE (up to 342 mg/L), but TCE concentrations drop off quickly and only DCE and VC are detected towards the edges of the plume.

## b. Microcosms: Cape Canaveral Air Station, FL

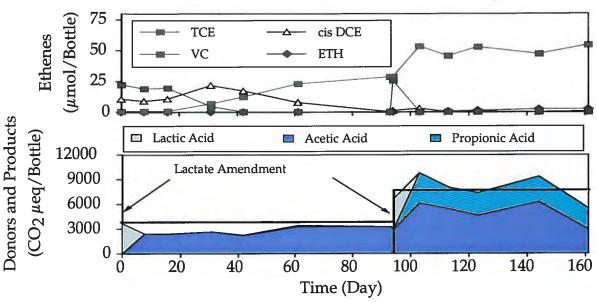
Microcosm studies at Cape Canaveral showed that all organic electron donors evaluated (lactate, butyrate, propionate, benzoate, yeast extract amendment) promoted enhanced dechlorination of the 2 mg/L TCE, 10 mg/L cDCE and 1.5 mg/L VC present in the site groundwater. Lactate was selected for the electron donor to be used in the field-testing.

The graphs below illustrate lessons learned from conducting microcosm studies. Upon the addition of lactate and vitamin  $B_{12}$  with no yeast extract, levels of TCE and cDCE show no significant signs of reduction. Alternatively, the addition of yeast extract along with lactate and vitamin  $B_{12}$  facilitated the onset of and completion of the dechlorination process.



CCAS Microcosm 5C-II (GPW09/10) Lactate + B<sub>12</sub>





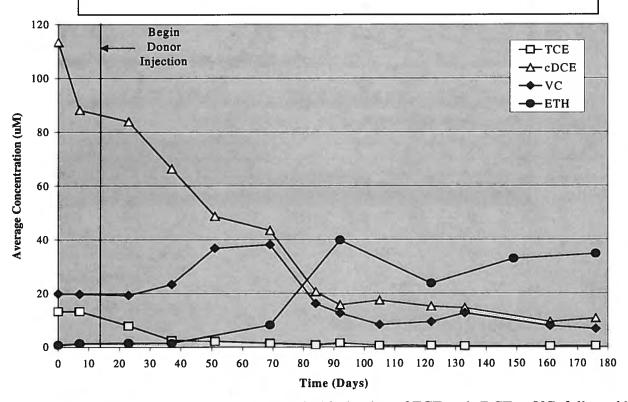
### c. Field Study: Cape Canaveral Air Station, FL

The standard RABITT design was modified for the site at Cape Canaveral Air Station in order to meet the State of Florida Underground Injection Control regulatory requirements. This regulation does not allow for reinjection of contaminated groundwater. The objective of the modified system was to allow for effective delivery and distribution of nutrients and electron donors and to provide for extensive monitoring and hydraulic control, without pumping groundwater aboveground. The modified system was installed at Facility 1381 in March 1999 and operated for six months.

The modified design consisted of two communicating wells, a series of 13 tri-level groundwater monitoring probes, and upgradient and downgradient monitoring wells. The system wells are a dual screen design, with one operating in an upflow mode and the other in a downflow mode. Each well was screened within two distinct zones (10-12.5 and 17.5-20 ft bgs). The wells are placed close enough to affect each other with the effluent from one well feeding the other. This results in groundwater circulation that can be used to mix and distribute the electron donor/nutrient formulation. Tri-level monitoring points were screened in three zones that covered similar depths and an intermediate zone. The monitoring probes were positioned around the treatment cell to provide three-dimensional data that was required to track the tracer and added electron donor/nutrients, calculate mass reductions during treatment, and evaluate gains and losses from the treatment cell through background groundwater migration. The monitored plot dimensions were 39 ft by 10 ft.

After initial tracer testing established the site hydrological conditions, lactic acid was injected into the communicating well system to maintain an initial groundwater concentration of 3 mM lactate. The total system pumping rate was approximately 2880 gal/day (7.6 L/ min).

# Cape Canaveral Air Station, Facility 1381 TCE and Daughter Products Average Chloroethenes



Cape Canaveral field-testing showed rapid dechlorination of TCE and cDCE to VC, followed by slower subsequent dechlorination to ethene under the established sulfate reducing to methanogenic conditions. Molecular probing indicated the presence of a dechlorinating organism similar to *Dehalococcoides ethenogenes*, an organism that has been shown to promote complete dechlorination with slow removal of VC. The treatment demonstrated reduction of TCE, cDCE, and VC by 88.7%, 90.6%, and 66.3%, respectively. The ethene concentration increased significantly to approximately 0.04 mM, but good molar balances were not possible due to diffusion. Overall, there was reasonable agreement between laboratory microcosm and field results.

### 2. Site #2: Naval Air Station Alameda, CA

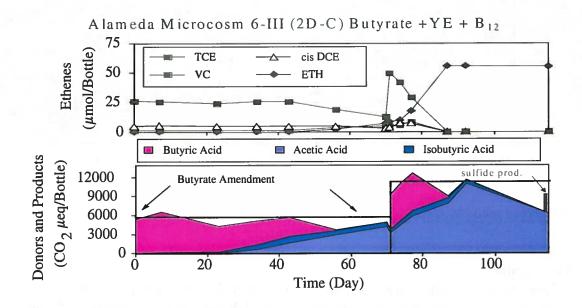
a. Site Description: Building 360 (Site #4) at Naval Air Station Alameda was selected for the second demonstration. This building has been used as an aircraft engine repair and testing facility, and consisted of former machine shops, cleaning areas, as well as plating and welding shops and parts assembly areas. Solvents used in the cleaning shop of Building 360 have included a mixture of 55% PCE and other chemicals such as dichlorobenzene, methylene chloride, toluene and 30-70% solutions of sodium hydroxide. Site characterization activities

performed by the facility revealed elevated levels of chlorinated solvents, primarily TCE (24 mg/L), DCE (8.6 mg/L) and VC (2.2 mg/L), between 5.5 and 15.5 feet bgs.

Depth to groundwater in the Building 360 area ranges between 4.4 feet and 6.5 feet bgs. Aquifer testing yielded hydraulic conductivity values from  $1.22 \times 10^{-3}$  to  $3.86 \times 10^{-3}$  cm/sec. The estimated groundwater flow is very low at only  $1.1 \times 10^{-5}$  cm/sec or 11.4 ft/year. It appears that groundwater in this area is very nearly stagnant.

### b. Microcosms: Naval Air Station Alameda, CA

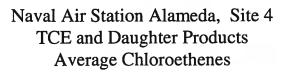
Microcosms showed that all electron donors tested but benzoate promoted enhanced dechlorination of TCE. Butyrate was chosen for field injection because of a shorter lag time associated with stimulating dechlorinating activity. TCE was rapidly dechlorinated to ethene under the established sulfate reducing to methanogenic conditions when supplied with a constant 3 mM supply of butyrate in the injected groundwater obtained from the supply well. Molecular probing to date has been negative for *D. ethenogenes*; however, recent data indicates that a closely related species may be present at the site.

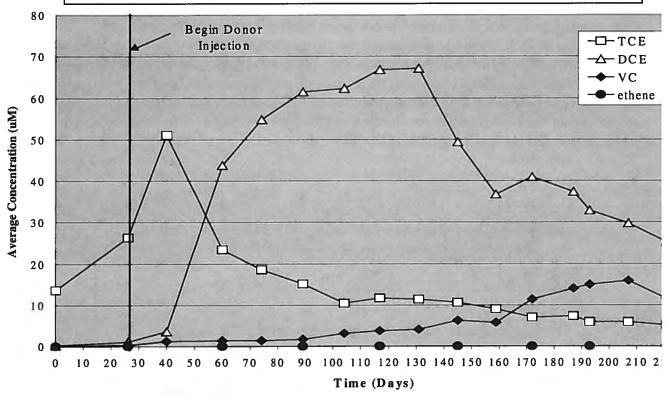


### c. Field Study: NAS Alameda

After baseline sampling and tracer testing, injection of butyric acid injection began at NAS Alameda June 1999 using a flow through system. The field test involved an upgradient injection well and downgradient extraction well with aboveground recirculation. The injection well was supplemented with TCE-contaminated groundwater from a separate supply well outside the influence of the 3-ft by 15-ft monitored plot. The injection, extraction and nine monitoring wells were all screened between 24 and 27 ft bgs. The total pumping rate for the system was 236 gal/day (0.62 L/min). Butyric acid and yeast extract were added to maintain initial in situ concentrations of 3mM and 20 mg/L respectively.

The treatment resulted in reductions of TCE and cDCE, of 94% and 82%, respectively, while VC remained at relatively low levels (maximum of 24% of cDCE level). Groundwater ethene levels continued to increase during the 6-month field test. Good agreement between microcosm and field results was also observed for the Alameda site.





### 3. Site #3: Fort Lewis, WA

### a. Site Description

The East Gate Disposal Yard (EGDY) covers approximately 29 acres at Ft Lewis, WA. Aerial photographs indicate that between 1940 and 1971 the EGDY was used as a storage and disposal site for various solid and liquid wastes. The photographic evidence shows that the wastes were disposed of in large trenches and pits and that, on occasion, the waste materials were burned. Waste materials disposed of at the EGDY include TCE and petroleum, oil, and lubricant wastes from equipment cleaning and degreasing activities conducted at the Fort Lewis Logistics Center.

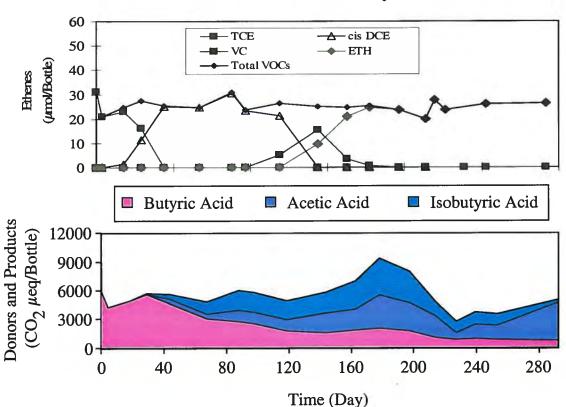
The depth to groundwater at the EGDY Site is approximately 10 feet bgs. Background groundwater velocities across the EGDY are in the range of 0.25 to 0.75 feet per day in the field test location. TCE, cDCE, VC, and BTEX constituents have been detected in groundwater samples from the EGDY Site. Of these, TCE and cDCE are most prevalent. Data from a

previous investigation indicate that reductive dechlorination may be occurring in the area but that the process is held up at cDCE.

### b. Microcosms: Ft Lewis, WA

Dechlorination in the Ft. Lewis microcosms was markedly slower than anticipated based on previous results with samples collected from Alameda Point and Cape Canaveral. The initial dose of TCE was removed from all of the amended, biotic reactors; however, formation of VC and complete conversion to ethene occurred in only a few bottles after 292 days of monitoring. The two amendments that did result in complete conversion to ethene in two of the three replicates were butyrate and high concentrations of yeast extract. Because the observed degradation of butyrate is slow, it appears to provide a relatively steady, long-term supply of electron equivalents for use.

The single, most important factor influencing dechlorination -- both lag and extent -- was probably the low native levels of TCE in the materials from which these microcosm sets were created. In the majority of bottles, there was a consistently long lag observed prior to the initiation of cDCE dechlorination. However, once cDCE dechlorination activity began, it was generally followed by concurrent transformation of VC to ethene. These patterns suggest that the transformation of TCE was mediated by different organisms than those responsible for cDCE and VC dechlorination. This type of pattern is consistent with the presence of a dechlorinating population in which *Dehaloccoides ethenogenes* is not the dominant member.



Ft. Lewis Microcosm 6-II Butyrate + YE +  $B_{12}$ 

### c. Field Study: Ft Lewis, WA

A conventional RABITT test system (shown in Figure 6.1 on page 54 of the draft RABITT protocol) was installed at Fort Lewis, with the exception that the gradient well was removed from the design based on the results of the tracer test and the measured gradient in the selected area. The three injection wells are spaced approximately 2 feet apart and the distances between the injection wells and each row of monitoring wells are 10 feet for a plot dimension of approximately 4 feet by 30 feet. A background monitoring well was installed upgradient of the plot to monitor any naturally occurring changes in background contaminant and geochemical profiles. An existing well in a contaminated area was used to provide the required supply of contaminated groundwater for injection into the test plot. The injected fluid imparts a gradient, which drives the flow of groundwater through the system. An existing pump-and-treat system recovers the injected water much further downgradient for any subsequent treatment that may be necessary.

### 4. Site #4: Marine Corps Base Camp Lejeune, NC

### a. Site Description

The contamination at Site 88, Marine Corps Base Camp Lejeune, occurred as a result of past operating procedures at the Base Dry Cleaners as well as due to leaking Underground Storage Tanks at the site. The surficial aquifer was encountered at depths of 6 to 15 feet bgs. The aquifer consists of a series of sediments, primarily sand and clay, which commonly extend to depths of 75 feet. The principal water supply for the base is found in the series of sand and limestone beds that occur between 50 and 300 feet bgs. This series of sediments generally is known as the Castle Hayne Formation, associated with the Castle Hayne Aquifer. The top of the Castle Hayne Aquifer was found at a depth of 40 to 60 feet bgs. Clay layers occur in both of the aquifers. However, the layers are thin and discontinuous in most of the area, and no continuous clay layer separates the surficial aquifer from the Castle Hayne Aquifer. Thin, discontinuous layers and lenses of silt, clay and/or peat are scattered throughout the sand. The hydraulic conductivity values estimated for the upper portion of the surficial aquifer ranged from 0.4 feet/day to 29.7 feet/day. The hydraulic conductivity values estimated for the lower portion of the surficial aquifer ranged from 56.4 feet/day to 85.5 feet/day.

## b. Microcosms: Camp Lejeune, NC

Core samples were taken from Camp Lejeune for microbial analysis in November 2000. Because previous site characterization indicated varying contaminant and geochemical profiles at increasing depths, two distinct microcosm sets were constructed. The first set was assembled using core material and groundwater from 15 to 19 feet bgs, while the second used core material and groundwater from 45 to 49 feet bgs. The construction of two microcosm sets was undertaken to more fully assess the potential for stimulating dechlorinating activity in the area. Transferring material between two microcosms from different depths should provide information about potential inhibitory conditions at the site, as well as an indication of the promise of

implementing a recirculating system in the field study. Monitoring of the Camp Lejeune microcosms is currently in-progress.

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